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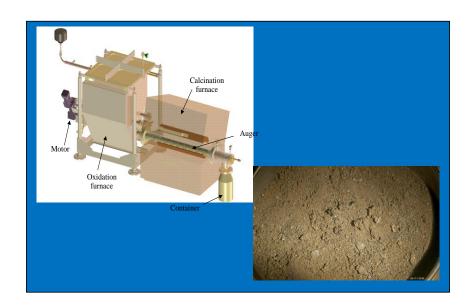
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ARIES Blend Lot Book Data Analysis

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1 Introduction

The Advanced Recovery Integrated Extraction System (ARIES) Program being executed at Los Alamos TA-55 produces unpolished plutonium oxide¹ powder from pit disassembly in batches of material referred to as a blend lot. Each blend lot is sampled and analyzed by Los Alamos group C-AAC (Chemistry Division, Actinide Analytical Chemistry Group) for radioactive and nonradioactive constituents contained in the material. A blend lot, which nominally consists of 12 kg(Pu), is then packaged into three DOE-STD-3013 containers that are analyzed by nondestructive analysis (NDA) to determine the fraction and total amount of plutonium mass relative to impure plutonium oxide. The analytical and NDA data are presented in a Blend Lot Book (BLB). The data are used to confirm that a blend lot grouping of three cans meets the requirements for off-site shipment specified in the formal interface control document (ICD).²

The analytical and NDA data from 49³ blend lots were reviewed to provide summary-level information on the constituents present in the blend lots. The results provide an overall perspective in historical amounts for constituent averages over all blend lots and similar information in the variation between constituent levels.

Note that to properly compare the can contents, it would be most appropriate to decay all of cans' constituents to a particular date. Previously, this analysis only examined statistical data as determined at the time of blend lot chemical analysis. The chemical analysis dates span the period from June 2011 to May 2015 (4 years), which is 28% of one Pu-241 half-life. Therefore, the approximate nature of the aggregate information must be properly understood in this light. However, to address this issue, a new table is included in this revision that decays the 48 blend lots to a single date.

2 Background

The oxide produced by the ARIES Program is prepared in batches of material intended for packaging into three DOE-STD-3013 containers. The Los Alamos procedural limit used for the amount of plutonium oxide quantity allowed per can is $4,800~g(PuO_2)$. This amount is just slightly smaller than the $5,000~g(PuO_2)$ limit specified in the 3013 standard and the ICD. Because processing of the plutonium oxide includes blending prior to packaging, an amount of plutonium sufficient to fill three containers is referred to as a blend lot. The 49 blend lots considered in this report include 146 cans of unpolished plutonium oxide.

Before packaging, representative samples are collected from each blend lot. The samples are analyzed by Los Alamos group C-AAC for radioactive and nonradioactive impurities, as well as the composition of plutonium isotopes. After packaging, cans are weighed to determine the total amount of oxide in an individual can. Nondestructive analysis (NDA) is then performed on each can to evaluate the total mass of plutonium and the fraction of plutonium relative to plutonium oxide. In this document, an "average lot can" amount is built on an analysis using the constituent average for all 49 blend lots. The term average lot can is also used if a constituent average, minimum, or maximum value is calculated using the average plutonium fraction or average plutonium mass derived from the average of all 146 cans in the 48 blend lots used. The variation in constituent levels is evaluated by analyzing minimum and maximum levels.

¹ Unpolished plutonium oxide means the powder includes impurities and is therefore not composed of pure plutonium oxide powder.

² "Los Alamos National Laboratory – Savannah River Site (K-Area Complex and/or Mixed Oxide Fuel Fabrication Facility) Plutonium Dioxide Powder Interface Control Document," Revision 2 (ICD-08-025-02, G-ESR-K-00039), prepared by Los Alamos National Laboratory (LANL) for NNSA Office of Fissile Materials Disposition, June 9, 2011. ³ BLB #1 was excluded from the radioactive constituents analysis because a different limit was used for maximum can quantity. Data were not available for BLB #3. The data for the 48 blend lots is derived from blend lot 2 and blend lots 4 through 50 reported in BLBs prepared from July 2011 through April 2015.

⁴ "Stabilization, Packaging, and Storage of Plutonium-Bearing Materials," DOE Standard, U.S. Department of Energy, DOE-STD-3013-2012, March 2012.

3 Radioactive Constituents Review

The Blend Lot Book (BLB) analytical data were reviewed for total amounts of plutonium and for radioactive isotopic contents. The results of the analyses are presented below.

3.1 Plutonium Oxide and Plutonium

The data for the amount of impure plutonium oxide and pure plutonium were reviewed for 48 blended lots, each containing three cans of material (except BLB #21, which only contained two cans). Summary data for an analysis covering 143 cans are presented below in Table 1. The data indicate that all cans are filled very closely to the limit of $4,800 \text{ g}(\text{PuO}_2)$. The data also show that the average amount of plutonium for an average lot can is 4169.2 g(Pu). An average lot can therefore contains approximately 87% pure plutonium relative to the total amount of unpolished oxide per container.

	Pu Oxide per Lot Can [g(PuO₂)/can]	Pu per Lot Can [g(Pu)/can]	Pu:Oxide Fraction [g(Pu)/g(PuO ₂)]
Average Lot Can	4799.5	4169.2	0.869
Maximum	4799.9	4229.3	0.881
Minimum	4797.8	4121.1	0.859
Standard Deviation	0.43	22.2	0.005

Table 1. Amounts of Plutonium and Plutonium Oxide per Lot Can for 48 Blend Lots.

Figure 1 presents data for the average, minimum, and maximum amounts of plutonium (per Table 1) for the set of 143 lot cans. The data indicate that the variation between the minimum and the maximum is just slightly more than 100 grams. The *average lot can* as indicated above contains 4169.2 grams.

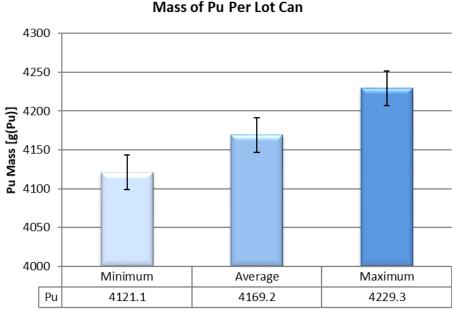


Figure 1. Minimum, average, and maximum amounts of plutonium in an average ARIES lot can.

The data for the amount of plutonium contained in each can is determined by calorimetry/gamma-ray spectroscopic analysis. Figure 2 provides the logical flow of how the average amount of plutonium for an "average lot can" is obtained. In subsequent sections, the term "average lot can" is used if the average amount of plutonium per lot can of 4169.2 g(Pu) is used to a calculate a constituent average, maximum, or minimum.

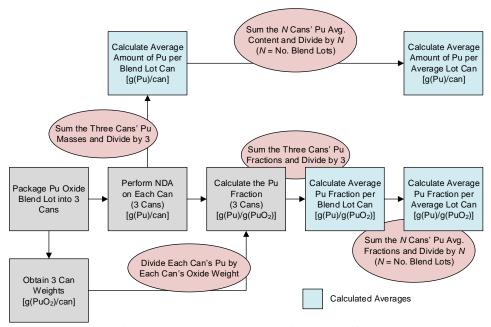


Figure 2. Method for obtaining average amount of plutonium for an average lot can.

3.2 Plutonium Isotopics

Representative samples sent to C-AAC are analyzed by thermal ionization mass spectrometry (TIMS) to obtain the weight percent of Pu-238, Pu-239, Pu-240, Pu-241, Pu-242, and Pu-244 in the sample relative to the total plutonium content (such that the sum of the isotopic fractions should equal 1). Figure 3 below presents the average weight percent of each isotope for the 48 lots analyzed. The results have been normalized to ensure that the sum of the plutonium isotopic fractions is mathematically equal to 1. The data show that Pu-239 is by far the largest constituent in the plutonium, and Pu-240 is the next most abundant, as expected for weapon-grade plutonium. Amounts of Pu-241, Pu-242, and Pu-238 are well below 1% of the plutonium mass. No data is shown for Pu-244 because the amounts are consistently found to be undetectable. Statistical summary data of the plutonium Isotopics is provided in Table 2 for the 48 blend lots.

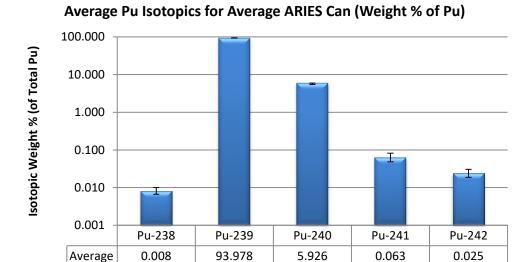
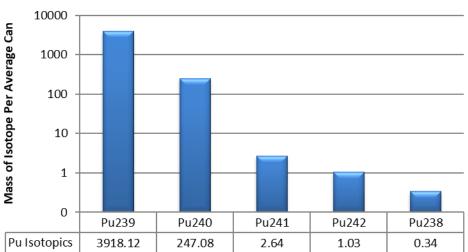


Figure 3. Average weight percent of plutonium isotopes per average lot can (note the logarithmic scale).

	Weight % of Pu Isotope for Average Lot Can [100 x g(Pu-X)/g(Pu)]					
	Pu-238 Pu-239 Pu-240 Pu-241 Pu-242					
Average	0.008	93.978	5.926	0.063	0.025	
Maximum	0.010	94.465	5.991	0.082	0.031	
Minimum	0.007	93.909	5.460	0.048	0.019	
Std. Dev.	0.001	0.079	0.074	0.009	0.003	

Table 2. Plutonium Isotopic Percentage [g(Pu-X)/g(Bulk Pu))] Statistics for 48 Blend Lots.

The mass of each isotope in an average lot can is calculated by multiplying each isotopic fraction by the plutonium amount in the average lot can. The results of this analysis are presented below in Figure 4 and Table 3. The data again show that Pu-239 is the predominant isotope. The other plutonium isotopes are present at amounts an order of magnitude smaller relative to Pu-239.



Pu Isotopics - Mass of Isotope Per Average Lot Can [g(Pu-X)/can]

Figure 4. Average plutonium isotopic masses [g(Pu-X)/can)] per average lot can (note the logarithmic scale).

	Mass of Pu Isotope for Average Lot Can				
	Pu-239 Pu-240 Pu-241 Pu-242 Pu-238				
Average	3918.12	247.08	2.64	1.03	0.34
Maximum	3971.66	253.25	3.45	1.29	0.42
Minimum	3873.58	226.67	2.01	0.78	0.28
Std. Dev.	20.11	3.61	0.39	0.12	0.03

Table 3. Plutonium Isotopic Mass [g(Pu-X)/can)] Statistics for an Average Lot Can.

3.3 Other Radioactive Impurities

The blend-lot samples are also analyzed to determine the other radioactive impurities. Thermal ionization mass spectrometry (TIMS) is used to evaluate the amount of uranium in the bulk plutonium. In general, uranium isotopics are not determined, but the majority of the uranium would be expected to be U-235 because of its parent's abundance (Pu-239). Radiochemistry is used to determine the amount of Am-241 (the decay product of Pu-241) and Np-237 (the decay product of Am-241) impurities. The results are reported in micro-gram (μ g) amounts relative to the bulk mass of plutonium in the sample. These results may, in turn, be converted to number of grams of impurity per average lot can (see Figure 5 for the calculation methodology). The data for the average, minimum, and maximum amounts of the minor radioactive impurities in an average blend lot can are shown below in Figure 6.

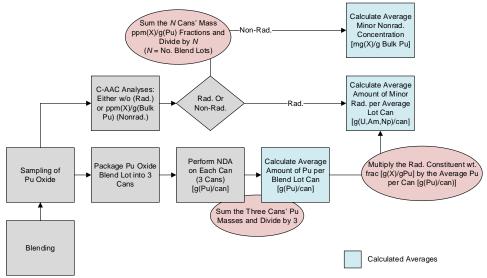
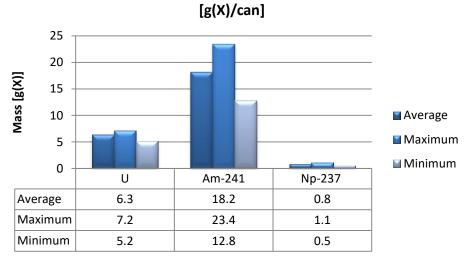


Figure 5. Methodology for calculating plutonium averages; methodology for calculating constituent mass per average lot can.

The data in Figure 6 show that gram-level quantities of radioactive impurities are present in the 48 blend lot cans. Am-241 is the most abundant radioactive impurity, followed by uranium and then Np-237. The data in Figure 6 show that more variation is associated with the Np-237 than for the uranium and americium, which is likely driven by the variability related to the small amount of the material. The data for the weight fractions of these three radioactive impurities, relative to plutonium mass, is provided in Table 4.



Mass of Radioactive Impurities per Average Lot Can

Figure 6. Radioactive impurity statistics for an average lot can.

Table 4. Radioactive Impurity Weight Fraction Statistics [$\mu g(X)/g(Pu)$] for an Average Lot Can.

	Wt. Fraction (ppm) Relative to Bulk Pu for Average Lot Can				
	U Am-241 Np-237				
Average	1523	4360	201		
Maximum	1720	5640	270		
Minimum	1230	3060	120		
Std. Dev.	122	456	39		

4 Nonradioactive Impurities

The blend-lot samples are analyzed for 41 nonradioactive⁵ chemical impurities from the 49 blend lots examined. The results are reported in microgram (μg) quantities relative to the mass of plutonium in the sample, or parts per million (ppm) relative to the bulk plutonium mass. Samples are analyzed by methods including inductively coupled plasma (ICP) atomic energy emission spectrometry (AES) and ICP mass spectrometry (ICP-MS) for metal impurities (aluminum and beryllium are analyzed by both ICP-AES and ICP-MS). Ion chromatography (IC) and other selected techniques are used for analyzing non-metallic impurities.

Figure 7 below provides data for the four highest chemical impurities in the lot data, which is derived simply by averaging the blend-lot data per Figure 5. A considerable amount of variation consists for many of the impurities including the four highest constituents shown in Figure 7. Figure 8 presents an example plot showing the amount of variation for chromium. Many other species show similarly pronounced variations. Where a constituent was below the detection limit, the listed detection limit was used in the calculations.

⁵ In these analyses, thorium is considered a nonradioactive element, which is appropriate given its 10-billion-year half-life

⁶ Recall that is unavailable BLB #3 and that BLB #1 was excluded from the analysis of the radioactive constituents; BLB #1 was, however, included in the analysis of the stable constituents.

Figure 9 provides data for all 41 nonradioactive impurities analyzed in the ARIES blend lots. A comparison of Figure 7 and Figure 9 show that gallium (Ga), nickel (Ni), and iron (Fe) are present in considerable abundance relative to the other impurities in the blend lots.

Table 5 provides the statistical data for all 41 nonradioactive constituents in the 49 blend lots and includes information on variation by referencing the standard deviation for each constituent.

Four Most Abundant Nonradioactive Impurities 12000 ug/g of Bulk Pu (ppm) 10000 8000 6000 ■ Average 4000 ■ Maximum 2000 ■ Minimum 0 Ga Ni Fe Cr ■ Std. Dev. Average 7573 765 452 144 Maximum 11000 3100 1000 290 Minimum 150 3700 270 66 Std. Dev. 1957 549 161.81

Figure 7. Highest four inorganic impurities in 49 ARIES blend lots.

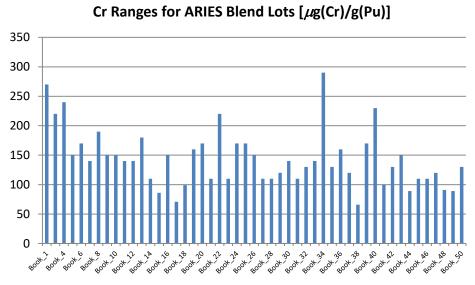
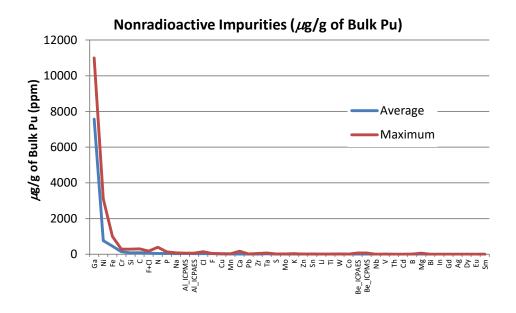


Figure 8. Example of amount of variation for nonradioactive impurities in blend lots.



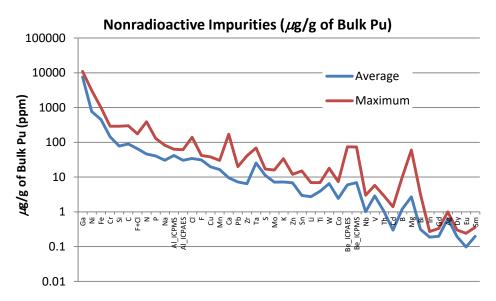


Figure 9. Trend data for 41 nonradioactive chemical impurities in 49 ARIES blend lots (linear and logarithmic scales).

Table 5. Summary of 42 Nonradioactive Impurities [$\mu g(X)/g(Bulk\ Pu)$] in 49 ARIES Blend Lots.

	Constituent	Avg.	Max.	Min.	Std. Dev.
1	Ga	7573	11000	3700	1957
2	Ni	765	3100	150	549
3	Fe	452	1000	270	162
4	Cr	144	290	66	48
5	С	90	300	23	49
6	Si	78	290	9.2	45
7	F+Cl	65	174	46	20
8	N	46	390	11	70
9	Al ICPMS	42	63	28	8.0
10	P	41	130	9.2	35
11	Cl	34	140	23	17
12	F	31	42	23	6.0
13	Al_ICPAES	30	61	11	12
14	Na	30	82	10	14
15	Та	26	69	15	8.6
16	Cu	20	38	5.9	8.0
17	Mn	17	30	8.1	4.2
18	S	11	17	5.4	2.3
19	Ca	9.5	170	1.0	24
20	К	7.2	34	4.6	4.4
21	Pb	7.2	20	3.2	3.4
22	Мо	6.9	16	3.4	3.1
23	Be ICPMS	6.9	73	0.35	15
24	Zn	6.8	12	4.6	1.9
25	W	6.5	18	2.6	3.5
26	Zr	6.4	41	1.4	7.1
27	Be_ICPAES	6.0	74	0.11	16
28	Ti	3.9	6.9	1.0	1.1
29	Sn	3.0	15	0.23	2.1
30	V	2.9	5.8	1.0	1.6
31	Li	2.7	6.9	1.0	1.2
32	Mg	2.7	60	0.23	8.6
33	Со	2.4	7.4	1.0	1.3
34	В	1.2	10	0.45	1.8
35	Th	1.0	2.9	0.3	0.5
36	Nb	1.0	3.0	0.4	0.6
37	Ag	0.63	1.0	0.46	0.16
38	Bi	0.31	3.2	0.22	0.43
39	Cd	0.30	1.4	0.12	0.22
40	Sm	0.20	0.36	0.11	0.06
41	Gd	0.20	0.33	0.09	0.06
42	Dy	0.20	0.30	0.11	0.06
43	In	0.19	0.27	0.07	0.06
44	Eu	0.10	0.24	0.01	0.04

5 Decayed Blend Lots

Another means of examining the pertinent data related to ARIES cans is to attempt to "normalize" the data by decaying the pertinent constituents to a particular date. In this case, we examine the data for BLB #2 through BLB #50. In the blend books, there are often several dates listed related to analysis of radioactive constituents. Whatever analysis dates were available, they were averaged to provide an estimated "birth" date for the blend lot. All lots were decayed to January 1, 2019.

To perform the decay, each lot's data set was accumulated into a single bulk material based on weight fractions. The main item of data that is missing in the blend books is a direct measurement of plutonium assay. Therefore, the assay was estimated by subtracting from 1 the weight fractions of the nonnuclear constituents and the uranium, americium, and neptunium. The plutonium isotopics were multiplied by the estimated assay to provide a weight fraction of the individual isotopes in the material. The uranium isotopics were estimated from a previous analysis where plutonium exchange data included uranium isotopics; the assumed weight fractions of the uranium isotopes are provided in Table 6.

Plutonium Parent	Uranium Isotope	Assumed wt. frac. [g(U-X)/g(U)]
Pu-238	U-234	0.0233
Pu-239	U-235	0.7789
Pu-240	U-236	0.180
Pu-242	U-238	0.0178

Table 6. Assumed Uranium Isotopics.

The decay calculation proceeds by converting the set of weight fractions into atomic fractions, as decay calculations are related to atomic constituents, not masses. In so doing, the material's atomic fractions are normalized to 1. The isotopes of plutonium and americium are decayed according to the appropriate equations, taking into account parent, daughter, and granddaughter amounts. The half-lives of Np-237 and uranium are long enough such that we consider these isotopes to be stable for the calculation. The nonnuclear atom fractions remain constant upon decay. After the decay calculation is performed, the material is reconverted back into a mass-based constituent mix.

The results of the decay calculation for the 48 cans examined is provided in Table 7. The statistical quantities shown are for the 48 blend lots, so the statistics related to the sums at the bottom of the table are the statistics related to the respective sum for each of the 48 lots.

Table 7. Statistical Results for 48 Blend Lots Decayed to Jan. 1, 2019 (all values are weight fractions).

Constituent	Average	Maximum	Minimum	Std. Dev.
Pu238	7.55 x 10 ⁻⁵	9.26 x 10 ⁻⁵	6.28 x 10 ⁻⁵	7.25 x 10 ⁻⁶
Pu239	0.924976	0.929341	0.921753	0.002070
Pu240	0.058298	0.058922	0.053678	0.000752
Pu241	0.000446	0.000563	0.000345	5.38 x 10 ⁻⁵
Pu242	0.000440	0.000303	0.000345	2.80 x 10 ⁻⁵
Am241	0.000242	0.005730	0.003229	0.000440
Np237	0.0004488	0.003730	0.000160	4.09 x 10 ⁻⁵
U234	3.97 x 10 ⁻⁵	4.47 x 10 ⁻⁵	3.29 x 10 ⁻⁵	2.50 x 10 ⁻⁶
U235	0.001366	0.001534	0.001159	8.44 x 10 ⁻⁵
U236	0.001300	0.000355	0.001133	1.95 x 10 ⁻⁵
U238	2.71 x 10 ⁻⁵	3.06 x 10 ⁻⁵	2.19 x 10 ⁻⁵	2.17 x 10 ⁻⁶
Ag	6.36 x 10 ⁻⁷	1.00 x 10 ⁻⁶	4.60 x 10 ⁻⁷	1.63 x 10 ⁻⁷
Al	3.56 x 10 ⁻⁵	6.15 x 10 ⁻⁵	1.95 x 10 ⁻⁵	8.68 x 10 ⁻⁶
В	1.07 x 10 ⁻⁶	1.00 x 10 ⁻⁵	4.50 x 10 ⁻⁷	1.57 x 10 ⁻⁶
Be	6.54 x 10 ⁻⁶	7.35 x 10 ⁻⁵	2.35 x 10 ⁻⁷	1.55 x 10 ⁻⁵
Bi	3.11 x 10 ⁻⁷	3.20 x 10 ⁻⁶	2.20 x 10 ⁻⁷	4.33 x 10 ⁻⁷
C	9.07 x 10 ⁻⁵	0.000300	2.30 x 10 ⁻⁵	4.92 x 10 ⁻⁵
Ca	9.45 x 10 ⁻⁶	0.000300	1.00 x 10 ⁻⁶	2.46 x 10 ⁻⁵
Cd	2.96 x 10 ⁻⁷	1.40 x 10 ⁻⁶	1.20 x 10 ⁻⁷	2.23 x 10 ⁻⁷
Cl	3.40 x 10 ⁻⁵	0.000140	2.30 x 10 ⁻⁵	1.73 x 10 ⁻⁵
Co	2.39 x 10 ⁻⁶	7.40 x 10 ⁻⁶	1.01 x 10 ⁻⁶	1.27 x 10 ⁻⁶
Cr	0.000141	0.000290	6.60 x 10 ⁻⁵	4.52 x 10 ⁻⁵
Cu	1.94 x 10 ⁻⁵	3.80 x 10 ⁻⁵	5.90 x 10 ⁻⁶	7.87 x 10 ⁻⁶
Dy	1.95 x 10 ⁻⁷	3.00 x 10 ⁻⁷	1.10 x 10 ⁻⁷	5.54 x 10 ⁻⁸
Eu	9.68 x 10 ⁻⁸	2.40 x 10 ⁻⁷	1.20 x 10 ⁻⁸	4.48 x 10 ⁻⁸
F	3.09 x 10 ⁻⁵	4.20 x 10 ⁻⁵	2.30 x 10 ⁻⁵	6.03 x 10 ⁻⁶
Fe	0.000440	0.000960	0.000270	0.000142
Ga	0.007600	0.011000	0.003700	0.00142
Gd	1.97 x 10 ⁻⁷	3.30 x 10 ⁻⁷	9.20 x 10 ⁻⁸	5.70 x 10 ⁻⁸
In	1.86 x 10 ⁻⁷	2.70 x 10 ⁻⁷	6.90 x 10 ⁻⁸	5.98 x 10 ⁻⁸
K	7.11 x 10 ⁻⁶	3.40 x 10 ⁻⁵	4.60 x 10 ⁻⁶	4.41 x 10 ⁻⁶
Li	2.63 x 10 ⁻⁶	6.90 x 10 ⁻⁶	2.30 x 10 ⁻⁶	1.02 x 10 ⁻⁶
Mg	2.59 x 10 ⁻⁶	6.00 x 10 ⁻⁵	2.30 x 10 ⁻⁷	8.69 x 10 ⁻⁶
Mn	1.62 x 10 ⁻⁵	2.70 x 10 ⁻⁵	8.10 x 10 ⁻⁶	3.71 x 10 ⁻⁶
Mo	6.94 x 10 ⁻⁶	1.50 x 10 ⁻⁵	3.40 x 10 ⁻⁶	2.86 x 10 ⁻⁶
N	4.67 x 10 ⁻⁵	0.000390	1.10 x 10 ⁻⁵	7.08 x 10 ⁻⁵
Na	2.93 x 10 ⁻⁵	8.00 x 10 ⁻⁵	1.10 x 10 1.00 x 10 ⁻⁵	1.14 x 10 ⁻⁵
Nb	9.95 x 10 ⁻⁷	3.00 x 10 ⁻⁶	3.60 x 10 ⁻⁷	5.54 x 10 ⁻⁷
Ni	0.000758	0.003100	0.000150	0.000552
P	4.01 x 10 ⁻⁵	0.000130	9.20 x 10 ⁻⁶	3.46 x 10 ⁻⁵
Pb	7.02 x 10 ⁻⁶	2.00 x 10 ⁻⁵	3.20 x 10 ⁻⁶	3.21 x 10 ⁻⁶
S	1.12 x 10 ⁻⁵	1.70 x 10 ⁻⁵	5.40 x 10 ⁻⁶	2.30 x 10 ⁻⁶
Si	7.72 x 10 ⁻⁵	0.000290	9.20 x 10 ⁻⁶	4.46 x 10 ⁻⁵
Sm	1.98 x 10 ⁻⁷	3.60 x 10 ⁻⁷	1.10 x 10 ⁻⁷	5.74 x 10 ⁻⁸
Sn	2.93 x 10 ⁻⁶	1.50 x 10 ⁻⁵	2.30 x 10 ⁻⁷	2.15 x 10 ⁻⁶
Ta	2.58 x 10 ⁻⁵	6.90 x 10 ⁻⁵	1.50 x 10 ⁻⁵	8.56 x 10 ⁻⁶
Ti	3.89 x 10 ⁻⁶	6.90 x 10 ⁻⁶	1.00 x 10 ⁻⁶	1.12 x 10 ⁻⁶
Th	1.04 x 10 ⁻⁶	2.90 x 10 ⁻⁶	2.60 x 10 ⁻⁷	5.15 x 10 ⁻⁷
V	2.91 x 10 ⁻⁶	5.80 x 10 ⁻⁶	1.00 x 10 ⁻⁶	1.63 x 10 ⁻⁶
Zn	6.81 x 10 ⁻⁶	1.20 x 10 ⁻⁵	4.60 x 10 ⁻⁶	1.94 x 10 ⁻⁶
W	6.53 x 10 ⁻⁶	1.80 x 10 ⁻⁵	2.60 x 10 ⁻⁶	3.50 x 10 ⁻⁶
Zr	6.20 x 10 ⁻⁶	4.10 x 10 ⁻⁵	1.40 x 10 ⁻⁶	7.05 x 10 ⁻⁶
-1	0.20 / 10	1120 % 10	1. 10 X 10	7.03 X 10
ΣΡυ	0.984038	0.988994	0.980495	0.002165
ΣU	0.001749	0.001964	0.001482	0.000109
Σnonrad.	0.001749	0.012378	0.005064	0.002056
TOTAL	1	1	1	0.002036
IOIAL	1		1	U